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NITROSAMINES

VOLUME 6

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INTRODUCTION

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Coverage of the subject has been restricted to that of Food Science and Technology Abstracts, which covers over 1200 of the important food journals, patents from 20 countries and books published world-wide. Every effort is made to include all significant references, but editorial discretion is used on the many articles of borderline interest. If the reader particularly needs an exhaustive search of the subject, we will be pleased to provide any other references that we have available. We would in any case encourage readers to write or telephone us with any comments or queries that they may have.

J. NEWTON,
ASSISTANT EDITOR

1

[Nitrosamines in foods. V. Distribution of secondary amines in foods. 2.]
Kawamura, T.; Sakai, K.; Miyazawa, F.; Wada, H.; Ito, Y.; Tanimura, A.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **12** (5) 394-398 (1971) [3 ref. Ja, en] [Yokohama City Inst. of Health, 2-17 Takigashira 1-chome, Isogo-ku, Yokohama]

A higher content of dimethylene was detected in whale meat than in mutton, chicken, pork and beef. Sausages consisting of pork, whale, mutton and tuna had a larger amount of dimethylamine than did bacon and ham. Modified powdered milk showed 5 times as much dimethylamine as did milk (approx. 0.12 vs. 0.025 μmol/g), while trace amounts of dimethylamine (<0.02 μmol/g) were found in butter and processed cheese. In the case of mackerel and cuttlefish, the boiled, roasted, canned or dried samples contained more secondary amines than did the raw samples. [See FSTA (1973) **5** 5B51-54 for parts I-IV.] TM

2

[Nitrosamines in foods. VI. Comparison of two extraction and determination methods of secondary amines.]
Ito, Y.; Sakuta, H.; Takada, H.; Tanimura, A.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **12** (5) 399-403 (1971) [4 ref. Ja, en] [Nat. Inst. of Hyg. Sci., 18-1, Kamiyoga, 1-chome, Setagaya-ku, Tokyo]

A comparative investigation was made of 2 methods for extraction and determination of secondary amines: one was the authors' method [see FSTA (1973) **5** 5B53] and the other was Dyer's method improved by Kawabata. Analytical results by the above 2 methods were agreeable with respect to secondary amines in 12 kinds of canned foods. TM

3

Effect of sodium nitrite and sodium nitrate on botulinal toxin production and nitrosamine formation in wieners.
Hustad, G. O.; Cerveny, J. G.; Trenk, H.; Deibel, R. H.; Kautter, D. A.; Fazio, T.; Johnston, R. W.; Kolari, O. E.
Applied Microbiology **26** (1) 22-26 (1973) [6 ref. En] [Oscar Mayer and Co., Madison, Wisconsin 53706, USA]

Wieners were formulated and processed approximating commercial conditions. 24 batches were made with 6 levels of $NaNO_2$ (0, 50, 100, 150, 200 and 300 μg/g), 4 levels of $NaNO_3$ (0, 50, 150 and 450 μg/g), and 2 levels of Clostridium botulinum (0 and 620 spores/g). After formulation, processing and vacuum packaging, portions of each batch were incubated at 27°C or held for 21 days at 7°C followed by incubation at 27°C for 56 days. The latter approximated distribution of product through commercial channels and potential temp. abuse at consumer level. Samples were analysed for botulinal toxin, nitrite, and nitrate after 3, 7, 14, 21, 28 and 56 days. When nitrite was not added, toxic samples were detected after 14 days at 27°C. At lowest level of nitrite, no toxic samples were observed until 56 days. Higher levels of nitrite completely inhibited toxin production throughout incubation. 9 uninoculated samples, representing various levels and combinations of nitrite and nitrate, were evaluated organoleptically. Flavour quality of wieners with nitrite was judged significantly higher ($P = 0.05$) than that wieners without nitrite. The 9 samples were negative for 14 volatile nitrosamines at a sensitivity level of 10 ng/g. Results indicated that nitrite effectively inhibited botulinal toxin formation at commercially employed levels in wieners and that detectable quantities of nitrosamines were not produced during preparation and processing for consumption. AS

4

Estimation of volatile N-nitrosamines by high-performance liquid chromatography.
Cox, G. B.
Journal of Chromatography **83** (1/2) 471-481 (1973) [10 ref. En] [Lab. of the Govt. Chemist, Cornwall House, Standford Street, London SE1 9NQ, UK]

Adaptations of 2 gas chromatographic methods (the reduction-amine derivative method (i) described in FSTA (1973) **5** 5A239 and the direct N-nitrosamine determination method (ii) used in FSTA (1973) **5** 1C22 and 1C23), used for determining N-nitrosodimethylamine, N-nitrosopyrrolidine and N-nitrosodimethylamine in foods, are described. Instead of using GLC, the samples are analysed by high pressure liquid chromatography using columns of Carbowax 400-Corasil or Carbowax 400-Silica Gel Si 60 (with 25% dioxan-2,2,4-trimethylpentane as mobile phase) or of Corasil C-18 (with 33% aqueous methanol) with method (i), and a column of di-(2-cyanoethyl) ether on Zipax (with 2,2,4-trimethylpentane as mobile phase) with method (ii). The methods were compared by analysing samples of pork luncheon meat and fried pigs liver. The results agreed well with those obtained by the gas chromatographic methods and were obtained in a shorter time. Method (ii) was shown to be more sensitive but more prone to interference from other extracted materials than method (i). MEG

5

[Nitrosamines in foods. VIII.]
Sakai, A.; Tanimura, A.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **12** (6) 485-488 (1971) [14 ref. Ja, en] [Nat. Inst. of Hygienic Sci., 18-1 Kamiyoga 1-chome, Setagaya-ku, Tokyo]

Qualitative analyses for nitrosamines were performed on 149 commercial food samples. Extraction by the modified Howard method and identification by TLC were used. Dimethylnitrosamine (DMNA) was detected in 6 of 20 samples of pressed ham and in 1 of 3 samples of hamburger. The detection limit was presumed to be approx. 15-25 parts/billion. DMNA and DENA (diethylnitrosamine) were also detected in 7 of 20 samples of salted salmon roe to which sodium nitrite had been added with the intention of improving the colour. TM

6

The effect of pH on dimethylnitrosamine formation in human gastric juice.
Lane, R. P.; Bailey, M. E.
Food and Cosmetics Toxicology **11** (5) 851-854 (1973) [17 ref. En] [Dept. of Food Sci. & Nutr., Univ. of Missouri, Columbia, 65201, USA]

The extraction and analytical procedure are described for determination of dimethylnitrosamine formed during the reaction for 1 h of a solution containing 200 ppm nitrite and 200 ppm dimethylamine with an equal vol. of human gastric juice at pH 1.7, 2.5, 3.5 or 4.5. From the preliminary results, it appears that the optimum pH for nitrosation of dimethylamine in human gastric juice is in the region of 2.5. VJG

7

Dimethylnitrosamine in Chinese marine salt fish.
Fong, Y. Y.; Chan, W. C.
Food and Cosmetics Toxicology **11** (5) 841-845 (1973) [14 ref. En] [Dept. of Biochem., Univ. of Hong Kong]

Different species of Cantonese salt fish (white herring, yellow croaker, anchovies, croaker, pomfret) were purchased from local retailers in the markets in Hong Kong and analysed for N,N-dimethylnitrosamine (DMNA). Concn of DMNA, residual nitrite, residual nitrate (ppm) respectively ranged as follows: white herring, 0.04-1.00, 1-2, 6-40; yellow croaker, 0.01-0.20, 1-4, 15-30; anchovies, 0.02-0.10, 1-2, 8-10; croaker, 0.02-0.03, 0-2, 10-20; pomfret, not detected, 1, 30. The DMNA was detected by GLC and confirmed in several samples by GLC-MS. DMNA levels in salt fish prepared with laboratory NaCl and market-bought crude salt respectively were (ppm): white herring, 0.01, 0.4; yellow croaker, 0.005, 0.2. Salt fish prepared with crude salt contained a higher concn. of DMNA. VJG

8

Formation of nitrosamines in a meat curing mixture.
Sen, N. P.; Miles, W. F.; Donaldson, B.; Panalaks, T.; Iyengar, J. R.
Nature, UK **245** (5420) 104-105 (1973) [11 ref. En] [Food Res. Lab., Health Protection Branch, Ottawa, K1A OL2, Canada]

Fairly high levels of nitrosopyrrolidine (≤0.105 ppm) and nitrosopiperidine (≤0.06 ppm) were detected in mettwurst produced in Canada. The source of these nitrosamines was found to be a dry curing mixture containing black pepper, paprika, 2 other spices, NaCl, $NaNO_3$ and $NaNO_2$ (0.96%). Spice-nitrite interaction under dry conditions was very slow; trials were, therefore, conducted with suspensions of 1 or 4% paprika or black pepper in solutions containing 20-2400 ppm $NaNO_2$. Results showed that nitrosopiperidine is formed predominantly from black pepper, and nitrosopyrrolidine from paprika. Further experiments showed that this curing mixture also contains approx. 0.85 ppm of N-nitrosodimethylamine. AJDW

9

A contaminant in N-nitrosodimethylamine confirmation by high resolution mass spectrometry.
Dooley, C. J.; Wasserman, A. E.; Osman, S.
Journal of Food Science **38** (6) 1096 (1973) [3 ref. En] [USDA E. Regional Res. Center, Philadelphia, Pennsylvania 19118, USA]

The carcinogenicity of N-nitrosamines poses a potential health hazard and therefore their presence in foods must be carefully validated and evaluated. Current indications are that N-nitrosamines, when found in foods, are present only in the μg/kg range, requiring precise methods for recovery and identification. The paper reports the possibility of confusing the identification of N-nitrosodimethylamine (DMNA) with the ^{13}C and ^{29}Si isotopes of trimethylsilyl ion when using high resolution MS and suggests that false positive identification of DMNA could possibly be avoided by one or more of the following: (1) utilizing GLC column coatings other than Carbowax M to achieve separation of DMNA from the silicon-containing compound; (2) use of alkali flame ionization detector to differentiate N-containing compounds; (3) low resolution MS analysis of fairly pure preparations to obtain the entire fragmentation pattern; and (4) monitoring 2 specific ion peaks for DMNA in the high resolution mode. IFT

10

[Cereal chemists' conference of the study group for cereal research held in Detmold on 22-24 May, 1973.] Getreidechemiker-Tagung 1973 der Arbeitsgemeinschaft Getreideforschung vom 22. bis 24. Mai in Detmold.
Germany, Federal Republic of,
Arbeitsgemeinschaft Getreideforschung eV

Mühle + Mischfuttertechnik **110** (25) 408-409; (29) 464-465 (1973) [De]

Summaries are given of the following papers presented at the conference: Modern methods for protein determination, by H. Bolling; Determination of protein in wheat and flour by the Rapid-Meter, by C. P. Greenwood; Evaluation of enzyme activity in malt flours with synthetic substrates, by J.-M. Brümmer; Studies of methods for determining grain hardness of wheat, by E. K. Meppelink; Nitrosamines in food, by K. Heyns; Observations on the growth of Aspergillus flavus on bread and the formation of aflatoxin, by G. Spicher; Texture of bread and bakery products, by L. Wassermann; Comparative evaluation of physical methods for measuring crumb consistency, by B. Thomas & A. Juretko; Wheat lipids and their enzymic oxidation, by A. Graveland; Enzymic decomposition of protein fractions and detection of fragments by "finger-priting", by H. D. Jodlbauer; and Effect of post-harvest treatment of domestic maize on lipid composition, by H. Bolling, P. Gerstenkorn, A. W. El Baya & H. Zwingelberg. RM

11

[Cereal chemists' conference 1973.]
Getreidechemiker-Tagung 1973.
Germany, Federal Republic of,
Arbeitsgemeinschaft Getreideforschung eV
Deutsche Lebensmittel-Rundschau **69** (10) 369-372 (1973) [De]

Abstracts are given of the following papers presented at this conference (held in Detmold, 22-24 May 1973): Texture of bread and bakery products, by L. Wassermann; Comparative evaluation of physical methods for evaluation of crumb consistency, by B. Thomas & A. Juretko; Nitrosamines in food, by K. Heyns; Growth of Aspergillus flavus on bread, and formation of aflatoxins, by G. Spicher; Effects of post-harvest treatment on the lipid composition of corn, by H. Bolling, P. Gerstenkorn, A. W. El Baya & H. Zwingelberg; Synthetic substrates for determination of enzyme activity in malt flours, by J.-M. Brümmer; Methods for determination of the hardness of wheat grains, by E. K. Meppelink; Wheat lipids and their enzymic oxidation, by A. Graveland; Determination of protein in wheat with the Research Association Indicator Device (Rapid-Meter), by C. P. Greenwood & D. J. Stevens; Modern methods for protein determination, by H. Bolling; Cereal processing information, by Baron von Weichs; Enzymatic determination of sugar and starch in bread and bakery products, by A. Menger; Enzymic degradation of protein fractions by Osborne's method, and analysis of the degradation products by the fingerprint technique, by H. D. Jodlbauer; Biosynthesis of wheat proteins, by W. Hertel; Determination of the biological value of proteins, by H. G. Ludewig; Study of the structure of gluten proteins, by M. Rohrlich; & Synthetic substrates for determination of protease activity in wheat, rye and their milling products, by D. Breyer & W. Hertel. [See also preceding abstr.] AJDW

12

[Formation of dimethylnitrosamine in salted Alaska pollack roe products and a preventive method.]
Kawabata, T.; Kurihara, M.; Kasai, E.; Yoshida, C.
Bulletin of the Japanese Society of Scientific Fisheries [Nihon Suisan Gakkai-shi] **39** (8) 883-889 (1973) [11 ref. Ja, en] [Dept. of Biomedical Res. on Food, Nat. Inst. of Health, Shinagawa-ku, Tokyo]

The formation of dimethylnitrosamine (DMNA) in salted Alaska pollack roe products when nitrite is applied as a colour fixative was studied. No DMNA was detected in products prepared with nitrite at concn. <0.9 millimoles; at higher concn. the addition of a mole-equivalent of sodium ascorbate to the sodium nitrite inhibited the formation of DMNA in the salted products. The freshness of the raw material did not influence the formation of DMNA in the salted products. [From En summ.] AA

13

Use of sodium ascorbate or erythorbate to inhibit formation of N-nitrosodimethylamine in frankfurters.
Fiddler, W.; Pensabene, J. W.; Piotrowski, E. G.; Doerr, R. C.; Wasserman, A. E.
Journal of Food Science **38** (6) 1084 (1973) [6 ref. En] [USDA E. Regional Res. Center, Philadelphia, Pennsylvania 19118, USA]

The effect of $NaNO_3$, sodium ascorbate (NaAsc) and sodium erythorbate (NaEry) on N-nitrosodimethylamine (DMNA) formation in frankfurters made with 150 or 1500 ppm $NaNO_2$ was investigated. $NaNO_3$, NaAsc or NaEry were added to the emulsion at their max. permissible levels and at 10 times these amounts. The frankfurters were cooked and smoked for either 2 or 4 h. Frankfurters prepared with 150 ppm $NaNO_2$ alone or with $NaNO_3$, NaAsc or NaEry had no DMNA. With 1500 ppm $NaNO_2$, which induced DMNA formation, $NaNO_3$ had no effect; however, use of NaAsc or NaEry prevented formation of DMNA in frankfurters processed for 2 h or reduced the amount formed after 4-h treatment. [See also FSTA (1973) **5** 1S57 & 11S1256.] IFT

14

Potential precursors of N-nitrosopyrrolidine in bacon and other fried foods.
Bills, D. D.; Hildrum, K. I.; Scanlan, R. A.; Libbey, L. M.
Journal of Agricultural and Food Chemistry **21** (5) 876-877 (1973) [6 ref. En] [Dept. of Food Sci. and Tech., Oregon St. Univ., Corvallis, 97331, USA]

The possibility of producing N-nitrosopyrrolidine from several compounds in a system designed to simulate the frying of fatty foods containing small amounts of water was investigated. With the

exception of tests on N-nitrosoproline, equimolar amounts of the compound to be tested and sodium nitrate were added to the system prior to heating. With a heat-up time of 10 min plus 10 min at 170°, N-nitrosopyrrolidine was produced from N-nitrosoproline, pyrrolidine, spermidine, proline, and putrescine in yields of 2.6, 1.0, 1.0, 0.4, and 0.04% theoretical, respectively. N-Nitrosopyrrolidine was not produced from glutamine, glutamic acid, and hydroxyproline. AS

15

[Analysis of N-nitrosamines by gas chromatography.]
Kawabata, T.; Matsui, M.; Ishibashi, T.; Nakamura, M.
Japan Analyst [Bunseki Kagaku] **21** (10) 1326-1332 (1972) [11 ref. Ja, en] [Dept. of Food Res., Nat. Inst. of Health, Shinagawa-ku, Tokyo]

Recently much attention has been paid to the possible formation of carcinogenic N-nitrosamines in foods. In the present study, chromatographic conditions for determination of N-nitrosamines, such as type of detector, column packing materials and carrier gas, were investigated and, in addition, qualitative and quantitative analyses for N-nitrosamines were conducted using the optimum chromatographic conditions. N-nitrosamines were determined in salted Alaska pollack which had been treated with sodium nitrite. [From En summ.] JA

16

Factors influencing the rate of formation of nitrosomorpholine from morpholine and nitrite. II. Rate enhancement in frozen solution.
Fan, T.-Y.; Tannenbaum, S. R.
Journal of Agricultural and Food Chemistry **21** (6) 967-969 (1973) [7 ref. En] [Dept. of Nutr. and Food Sci., Massachusetts Inst. of Tech., Cambridge, 02139, USA]

The reaction of nitrite and morpholine as a model of a nitrosation reaction has been studied in frozen model systems and in frozen milk. The rate was considerably enhanced in frozen systems compared to that expected for a supercooled solution at the same temp. The degree of enhancement appeared to be a function of overall solute concn., which in turn controls the amount of unfrozen solution at any temp. Because this type of nitrosation reaction is third-order, a 3-fold concn. of reactants leads to a 27-fold increase in reaction rate. A second type of rate enhancement was caused by selective precipitation of sodium phosphate buffers, leading to an increase in the acidity of the solvent and a higher reactant concn. The pH changes under these conditions were determined through the use of indicator dyes. Experiments in frozen milk show the results to be applicable to foods. [See FSTA (1973) 5 8C233 for previous part.] AS

17

Natural inhibition of nitrosation reactions: the concept of available nitrite.
Fan, T. Y.; Tannenbaum, S. R.
Journal of Food Science **38** (6) 1067-1069 (1973) [18 ref. En] [Dept. of Nutr. & Food Sci., Massachusetts Inst. of Tech., Cambridge, 02139, USA]

Different ratios of ascorbic acid/nitrite were used to study the rate of nitrosation of morpholine in a model system. Above a ratio of 2:1, the formation of nitroso-morpholine was completely inhibited by the ascorbic acid. The nitrosation of morpholine was also studied in a 5 or 10% reconstituted dried milk system at pH 6. Compared with a rate of nitrosation in buffer solution at pH 6 of 48 μM/h, that in the milk systems was only 12 and 24 μM/h in 5 and 10% dried milk respectively. Ultrafiltration experiments showed that the nitrite was not bound to the milk proteins. The results indicate that nitrites may be bound in foods in several complex forms, not all of which are available for nitrosation of amines but which may be measured by the Griess reaction. MEG

18

Group analysis of volatile and non-volatile N-nitroso compounds.
Fine, D. H.; Rufeh, F.; Lieb, D.
Nature, UK **247** (5439) 309-310 (1974) [11 ref. En] [Thermo Electron Corp., 85 First Avenue, Waltham, Massachusetts 02154, USA]

Various volatile nitrosamines, suspected to be carcinogenic, have been found in cured meats and fish. Analytical methods are available for 14 of these if present in the food at μg/kg levels; the procedures are lengthy and involve concentration, distillation, extraction, GLC separation and detection by Coulson conductivity or flame ionization GLC. This is not specific, and final confirmation by GLC-MS is essential. There are no methods yet available for non-volatile nitrosamines of high mol. wt. An apparatus is described which is specific for N-nitroso compounds and capable of detection at the μg/kg level without preconcentration and purification. The N-nitroso compound is dissolved directly from the food in dichloromethane, injected into a flash heater, and swept by argon carrier into a catalytic pyrolysis chamber where a tungsten oxide catalyst selectively splits the nitroso compounds. The products pass to a reaction chamber at reduced pressure (3.5 torr) to which O_3 is admitted and reacts with nitric oxide to give excited nitrogen dioxide; the latter rapidly decays with light emission at 0.6-2.8 μm. A photomultiplier detects and measures the intensity of light emission, which is directly proportional to the mol of nitroso compound. The instrument is sensitive to 0.4×10^{-12} mol nitroso compound and responds to both volatile and non-volatile types; no false positive have been obtained with a wide

variety of closely related compounds. Recoveries (method given) of 75-100% of additions of volatile (20-232 µg/kg) and non-volatile (60-120 µg/kg) nitroso compounds to fresh beef and herring were obtained with a single extraction. ELC

19

[Gas chromatographic screening of nitrosamines in foods with the nitrogen flame ionization detector.]
Gaschromatographisches Screening von Nitrosaminen in Lebensmitteln mit dem Stickstoff-Flammenionisationsdetektor.
Riedmann, M.
Journal of Chromatography **88** (2) 376-380 (1974) [6 ref. De] [Hewlett-Packard GmbH, Gutenbergstr. 6, D-7501 Grötzingen, Federal Republic of Germany]

To determine small concn. of nitrosamines in foods it is necessary to employ preliminary extraction, clean-up and concn. procedures followed by selective GLC. The GLC is combined with a selective detector for final confirmation of the presence of nitrosamines; this may be in the form of an electron capture detector (ECD), a mass spectrometer (MS) or a nitrogen flame ionization detector (NFID). Problems may arise from the presence of interfering substances naturally present in foods, often in higher concn. than the nitrosamines. Disadvantages of the ECD and MS methods are discussed; it is considered that these methods do not always ensure positive identification of nitrosamines, for which the NFID is preferable. A gas chromatogram is given to illustrate the sensitivity of detection of di-n-amylnitrosamine extracted from wheat flour and its clear separation from 5 impurities with a NFID detector. Anothe chromatogram shows the peaks obtained with a methylene chloride extract of nitrosamines from a dairy product, due to dimethyl-, diethyl-, and di-n-butylnitrosamines, 1-nitrosopiperidine, an organic P compound and 3 impurities containing no N. The NFID is highly selective and also sensitive to very small concn. of nitrosamines. A content of 10-20 pg nitrosamine at the GLC column can be estimated with an accuracy of ±10%. For screening analysis about 10 g of enriched material is adequate for 1 µg/kg nitrosamine concn. Results are not affected significantly by 1000-fold concn. of non-N impurities. ELC

20

[The importance of food research chemists in environmental protection.] Die Bedeutung des Lebensmittelchemikers aus dem Forschungsbereich für den Umweltschutz.
Fresenius, W.
Deutsche Lebensmittel-Rundschau 70 (1) 13-15 (1974) [14 ref. De] [Inst. Fresenius, Chem. & Biologische Lab. GmbH, 6200 Wiesbaden, Kapellenstrasse 11-15, Federal Republic of Germany]

Problems of the contamination of foods and water with aflatoxins, heavy metals, biocides, nitrosamines and polycyclic aromatic hydrocarbons are discussed, with special reference to sampling methods and analytical techniques. AJDW

21

Formation of dimethylnitrosamine from dimethylamine and trimethylamine at elevated temperatures.
Scanlan, R. A.; Lohsen, S. M.; Bills, D. D.; Libbey, L. M.
Journal of Agricultural and Food Chemistry **22** (1) 149-151 (1974) [14 ref. En] [Dept. of Food Sci. and Tech., Oregon St. Univ., Corvallis, 97331, USA]

22

[Nitrosamines in Italian meat products.]
Cantoni, C.; Acqua, V. I'; Renon, P.
Industrie Alimentari **13** (1) 97-101, 104 (1974) [38 ref. It]

Contents of nitrate, nitrite and nitrosamines were determined in many samples of meat products, including 20 mortadella sausage, 20 canned meat, 40 Milan ham, 20 chopped pork, 20 luncheon meat, 40 Milan salami, 30 liver sausage, 20 cooked shoulder, 20 cooked ham, 10 cooked tongue and 10 small sausage. Data are tabulated of max. and min. values found and of % of nitrosamine-positive samples. Nitrosamines could not be detected in the Milan hams, chopped pork or luncheon meat. When found in other products the quantities were minute and much below a possible toxic level; the max. individual figure recorded was 0.119 mg/kg in a single sample of cooked shoulder and other max. values ranged from 0.025 to 0.078 mg/kg. ELC

23

[Problems of colour in cooked hams.]
Cantoni, C.; Maccapani, M.; Calcinardi, C.
Industrie Alimentari **13** (1) 81-89 (1974) [23 ref. It]

A preliminary discussion is presented on factors influencing description of colour (tint, intensity, brightness), methods of measurement (transmission, reflectance), arbitrary colour units, and the chemical roles of myoglobin, oxymyoglobin, metmyoglobin, globins, nitrate and nitrite and Fe^{2+} and Fe^{3+} in meat pigmentation. Samples were examined from 6 samples of cured cooked shoulder and 11 manufactured hams, in each case including 4 different muscles, and using fresh meat as controls; all were from Large White pigs of the same wt. Detailed results are tabulated for total pigments, nitroso pigments, free haem groups, nitrate and nitrite contents and values at different depths in the same muscle, together with

analyses of variance for each pigment group. The analytical method of Hornsey [J. Sci. Food Agric. (1956) 7, 534] was used. Significant differences in contents of total pigments, nitroso pigments and free haems were found between shoulder meat and prepared hams, between different muscles of the same sample, and between different layers of the same muscle. ELC

24

[The problem of nitrosamine formation by the reaction of monosaccharides with amino acids (Maillard reaction) in the presence of sodium nitrite. II.] Zur Frage der Entstehung von Nitrosaminen bei der Reaktion von Monosacchariden mit Aminosäuren (Maillard-Reaktion) in Gegenwart von Natriumnitrit. II.
Heyns, K.; Röper, H.; Koch, H.
Zeitschrift für Lebensmittel-Untersuchung und -Forschung **154** (4) 193-200 (1974) [18 ref. De, en] [Inst. für Organische Chemie und Biochemie, Univ., Hamburg, Federal Republic of Germany]

3 model mixtures (D-glucose/DL-alanine, D-glucose/L-lysine and D-glucose/L-glutamic acid) were browned under Maillard conditions. Sodium nitrite was added either before or after browning. When the nitrite was applied after browning, a second browning reaction occurred. Reaction conditions (pH and browning temp.) were varied. By GLC and GLC/MS analysis of stable alkaline volatile Maillard products, formation of alkylated pyrazines, furan and pyridine derivatives was confirmed. Thus nitrosopiperidine was detected on addition of sodium nitrite after browning of D-glucose/L-lysine mixtures. A one unit change of pH and browning temp. in the range 100-150°C do not influence the qualitative composition of volatile Maillard products for a certain model mixture. Slight differences occurred in the quantitative composition. [See FSTA (1971) **3** 7C124 for previous part.] AS

25

[Studies on secondary amines in foods. II. Differentiation and determination of nitrosated secondary amines by GLC.]
/Kawabata, T.; Ishibashi, T.; Matsui, M.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **14** (1) 37-44 (1973) [14 ref. Ja, en]

Volatile secondary amines in foods can be differentiated and determined as follows. Secondary amines in a food sample are extracted with trichloroacetic acid and distilled in a vacuum under alkaline conditions. Sodium nitrite is reacted with the amines in the distillate. The nitrosamines formed are extracted with methylene chloride, concentrated and determined in a gas chromatograph equipped with a modified thermionic detector. 2 types of column (Chromosorb W-25% PEG 6000 and Chromosorb W-20% Versamid 900) are employed for GLC; the former is suitable for quantitative analysis of nitrosated secondary amines, the recovery of which is 95%. In one application of this method, it was established that dimethylamine is the major secondary amine in salted Alaska pollack and "fish ham". TM

26

[Nitrosamines in foods. X. Relation between alkalinity and recovery in the determination of secondary amines.]
Ayukawa, I.; Sakai, A.; Tanimura, A.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **14** (1) 100-104 (1973) [4 ref. Ja, en] [Nat. Inst. of Hygienic Sci., Kamiyoga 1-chome, Setagaya-ku, Tokyo]

An objection has been raised to the steam distillation stage with 10N NaOH solution in the authors' colorimetric method for determination of dimethylamine in foods, because dimethylamine may be formed from monomethylamine, trimethylamine or trimethylamine oxide contained in the foods. Experiments studying this problem did not show any inferiority in the authors' method. Use of magnesium oxide instead of NaOH was also investigated. [See FSTA (1974) **6** 4C135 for part IX.] TM

27

Stability of ascorbate in bacon.
Newmark, H. L.; Osadca, M.; Araujo, M.; Gerenz, C. N.; Ritter, E. de
Food Technology **28** (5) 28, 30-31, 60 (1974) [24 ref. En] [Product Development Dept., Hoffmann-La Roche Inc., Nutley, New Jersey 07110, USA]

Since sodium ascorbate can block the formation of nitrosamines from the reaction of $NaNO_2$ and susceptible amines, the stability of sodium ascorbate was determined in bacons processed and vacuum packed under commercial conditions with various levels of sodium ascorbate. The effects of storage in the freezer and refrigerator in both unopened and opened packages and of subsequent frying on the stability of ascorbate were measured. Optimum stability of sodium ascorbate in these bacons was obtained by storage in the freezer. IFT

28

Effect of frying and other cooking conditions on nitrosopyrrolidine formation in bacon.
Pensabene, J. W.; Fiddler, W.; Gates, R. A.; Fagan, J. C.; Wasserman, A. E.
Journal of Food Science **39** (2) 314-316 (1974) [13 ref. En] [USDA, E. Regional Res. Center, Philadelphia, Pennsylvania 19118, USA]

Nitrosopyrrolidine (NO-Pyr) formation in bacon is primarily dependent on frying temp. and not time. Cooking methods affect the amount of NO-Pyr formed: pan frying produces the highest level of NO-Pyr with variable concn. formed on baking, broiling and cooking in a 'baconer'. Microwave

oven treatment produced the lowest amount of NO-Pyr. A model system study of the decarboxylation of nitrosoproline shows this precursor, which may be present in bacon, is maximally converted to NO-Pyr at 185°C, near the recommended temp. for frying. IFT

29

Ascorbic acid and biological alkylating agents.
Edgar, J. A.
Nature, UK **248** (5444) 136-137 (1974) [23 ref. En] [CSIRO Div. of Animal Health, Private Bag No. 1, PO, Parkville, Victoria 3052, Australia]

In this article the author suggests a mechanism whereby ascorbic acid reacts with alkylating agents with the formation of a C-C bond which is not readily susceptible to hydrolytic cleavage, e.g. ascorbigen is formed by the reaction of the ascorbic acid anion with indole-3-methanol. Alkylation of ascorbic acid effectively competes with that of nucleic acid and proteins, and this competition may be the reason for the observed protective effect of sodium ascorbate on the hepatotoxicity of dimethylnitrosamine. Rapid depletion of cellular ascorbic acid by this alkylation process will also have an adverse effect on a number of cellular processes. MEG

30

A rapid method for the semi-quantitative determination of volatile N-nitrosamines in alcoholic beverages.
Castegnaro, M.; Pignatelli, B.; Walker, E. A.
Analyst **99** (1176) 156-162 (1974) [7 ref. En] [Unit of Environmental Carcinogens, Int. Agency for Res. on Cancer, 150 Cours Albert Thomas, 69008 Lyon, France]

The paper describes a method for rapid extraction of N-nitrosamines with methylene chloride from strong alcoholic drinks after saturating the water-ethanol phase with magnesium perchlorate. The nitrosamines are then determined by oxidation to nitramines and clean-up of the oxidation products by adsorption chromatography on a column containing 2 layers of different grades of alumina, followed by gas chromatography with an electron-capture detector. Recoveries of 10 µg/kg each of 7 nitrosamines added to aqueous ethanolic solution were recorded. Recoveries were good, with the exception of nitrosodimethylamine and nitrosopyrrolidine. The method could also be used for low-alcohol beverages, such as beer. PG

31

Formation of N-nitrosamines in low moisture systems.
Gray, J. I.; Dugan, L. R., Jr.
Journal of Food Science **39** (3) 474-478 (1974) [31 ref. En] [Dept. of Food Sci. & Human Nutr., Michigan St. Univ., East Lansing, 48823, USA]

Experiments have shown that N-nitrosamines can be formed when sodium nitrite and dialkylamines are heated in low-moisture model systems. Parameters which may influence the reaction were investigated. Results revealed that max. N-nitrosamine formation depended on temp. and time of heating, sodium nitrite-amine ratio and pH of the system. IFT

32

[The safety of marine fisheries products. III. Dimethylnitrosamine.]
Nakamura, M.; Usuki, M.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **14** (3) 264-271 (1973) [16 ref. Ja] [Hokkaido Central Fisheries Expt. Sta., Yoichi-cho, Hokkaido, Japan]

The diethylnitrosamine content of commercial marine fisheries products was investigated; diethylnitrosamine was detected in 14 of 65 samples tested. The relationship between diethylnitrosamine and trimethylamine oxide concn. in fishery products is discussed. TM

33

Effect of sodium nitrite concentration on the formation of nitrosopyrrolidine and dimethylnitrosamine in fried bacon.
Sen, N. P.; Iyengar, J. R.; Donaldson, B. A.; Panalaks, T.
Journal of Agricultural and Food Chemistry **22** (3) 540-541 (1974) [15 ref. En] [Food Res. Lab., Health Protection Branch, Dept. of Health and Welfare, Ottawa, Ontario K1A OL2, Canada]

Fried bacon samples prepared with 0, 50, 100, 150 and 200-ppm levels of $NaNO_2$ were analysed for nitrosopyrrolidine and dimethylnitrosamine. The samples prepared without any added nitrite were negative, but all the fried samples with added nitrite contained detectable levels (2-20 parts/billion) of nitrosamines. The levels of nitrosopyrrolidine correlated well with the initial concn. of nitrite but not with that of nitrite found in the raw bacon just prior to frying. The identity of nitrosopyrrolidine in a few samples was confirmed by GLC-MS. AS

34

[Mutagenic effect of nitrosomethylguanidine on the biosynthesis of free amino acids from Brevibacterium flavum.]
Murgov, I.; Keskinova, D.; Yanakieva, N.
Nauchni Trudove, Vissh Institut po Khranitelna i Vkusova Promyshlennost **19** (3) 311-317 (1972) [9 ref. Bg, ru, en]

35

[Nitrosamines in foods.] Über Nitrosamine in der Nahrung.
Heyns, K.
Veröffentlichung, Arbeitsgemeinschaft Getreideforschung eV No. 150, 45-54 (1973) [26 ref. De]

The occurrence of carcinogenic nitroso compounds (especially nitrosamines) in foods is discussed, with reference to literature data. Aspects covered include: reactions leading to formation of nitrosamines; the concn. of nitrosamine precursors (nitrites, nitrates and secondary amines) in various foods; the biological mechanism of the carcinogenic activity of nitrosamines; and methods of quantitative analysis of nitrosamines. AJDW

36

[Report on the Cereal Chemistry Conference, 22-24 May 1973.] Bericht über die Getreidechemiker-Tagung vom 22.-24. Mai 1973.
Germany, Federal Republic of,
Arbeitsgemeinschaft Getreideforschung eV (Wassermann, L.; Spicher, G.; Thomas, B.; Juretko, A.; Heyns, K.; Bolling, H.; Gerstenkorn, P.; El-Baya, A. W.; Zwingelberg, H.; Greenwood, C. T.; Stevens, D. J.; Meppelink, E. K.; Graveland, A.)
Veröffentlichung, Arbeitsgemeinschaft Getreideforschung eV No. 150, 191pp. (1973) [De]

The full text is given of the following papers presented at this conference, held under the auspices of the Society for Cereal Research and the Federal Cereal Processing Research Institute: Texture of bread and bakery products, by L. Wasserman (pp. 3-15, 33 ref.); Growth and aflatoxin formation by Aspergillus flavus on bread, by G. Spicher (pp. 17-27); Comparative studies on physical methods for determination of the consistency of bread, by B. Thomas & A. Juretko (pp. 29-44, 23 ref.); Nitrosamines in foods, by K. Heyns (pp. 45-54, 26 ref.); Effects of post-harvest treatments on the fat composition of corn, by H. Bolling, P. Gerstenkorn, A. W. El-Baya & H. Zwingelberg (pp. 55-61, 8 ref.); Modern methods for determination of protein, by H. Bolling (pp. 63-73, 7 ref.); Determination of protein in wheat and flour with the Research Association Protein Indicator Device (Rapid-Meter), by C. T. Greenwood & D. J. Stevens (pp. 75-80); Determination of the hardness of wheat grains, by E. K. Meppelink (pp. 81-93, 26 ref.); Wheat lipids and their enzymic oxidation, by A. Graveland (pp. 95-102, 7 ref.).
AJDW

37

[Factors influencing the formation of nitrosamines and their toxic effects.]
Polic, M.
Tehnologija Mesa 14 (12) 395-396 (1973) [8 ref. En, sh] [Jugoslavenski Inst. za Tehnologiju Mesa, Belgrade, Yugoslavia]

Factors influencing nitrosamine formation (nitrite concn., secondary amine concn., pH, temp., reaction time and presence of protective colloids, e.g. starch) are discussed; the presence of nitrites is necessary for nitrosamine formation in meat products. It is not yet certain whether the residual nitrite concn. permitted in meat products is sufficient to result in nitrosamine formation. STI

38

[Studies on N-nitrosamines in fishery products. I. Determination of trace quantities of volatile N-nitrosamines by thin-layer and gas-liquid chromatography.]
Kawabata, T.; Nakamura, M.; Matsui, M.; Ishibashi, T.
Bulletin of the Japanese Society of Scientific Fisheries [Nihon Suisan Gakkai-shi] 40 (1) 79-85 (1974) [14 ref. Ja, en] [Dept. of Biomed. Res. on Food, Nat. Inst. of Health, Shinagawa-Ku, Tokyo, Japan]

The determination of trace quantities of volatile N-nitrosamines by TLC and GLC was assessed. It was found that the maximum rate of colour development of dimethylnitrosamine (DMNA) on a TLC plate was obtained by UV irradiation. Resolutions and sensitivites of a gas chromatograph equipped with an alkali thermionic detector (KBr crystal) for 8 N-nitrosamines were compared with 4 types of column; Chromosorb W-PEG 6000 and Chromosorb W-Versamid 900 were found to be suitable for the qualitative and quantitative analyses of trace amounts of N-nitrosamines. The formation of DMNA in nitrite-treated Alaska pollack roe was confirmed by GLC-MS. [From En summ.] [See also following abstr.] AA

39

[Studies on N-nitrosamines in fishery products. II. Methods for extraction and clean-up test solutions containing N-dimethylnitrosamine to be analyzed by gas-liquid chromatography.]
Kawabata, T.; Nakamura, M.; Matsui, M.; Ishibashi, T.
Bulletin of the Japanese Society of Scientific Fisheries [Nihon Suisan Gakkai-shi] 40 (1) 87-96 (1974) [9 ref. Ja, en]

Methods for the extraction and clean-up of test

solutions containing N-dimethylnitrosamine (DMNA) to be analysed by GLC were assessed and a series of recovery tests of DMNA added to fish samples were carried out. It was found that DMNA could be extracted with dichloromethane under alkaline (K_2CO_3) conditions when the extraction and separation by centrifugation were repeated 3 times at room temp. However, recovery of DMNA added to fish samples was found to be only 60%; it is suggested that the reason for the low recovery rate might be loss of DMNA during the 3 concentration steps of dichloromethane extracts under vacuum. [See also preceding abstr.] [From En summ.] AA

40

[γ-Radiolysis of dimethylnitrosamine.]
Hirano, T.
Food Irradiation [Shokuhin-Shosha] **8** (1) 70-77 (1973) [11 ref. Ja, en] [Dept. of Food Sci. and Tech., Univ. of Fisheries, Minato-ku, Tokyo, Japan]

Considerable attention is currently being focused on dimethylnitrosamine (DMNA), which, under certain conditions, can be formed in seafoods. Studies were made of γ-radiolysis of DMNA in aqueous solutions and in homogenates of seafoods. After irradiating 100, 250 and 500 ppm DMNA with doses of 0.3-6.0 Mrad, residual DMNA was determined spectrophotometrically and polarographically. Amounts of nitrite and nitrate ions formed from irradiated DMNA were determined colorimetrically with Griess-Romijin reagent. From the UV absorption spectra shift in model systems containing $NaNO_2$ and KNO_3 and the formation of nitrite and nitrate ions from irradiated DMNA, it is assumed that the nitroso group of DMNA is readily oxidized by irradiation. Although the effect of pH on the radiolysis of DMNA in McIlvaine buffer solutions was negligible at pH 3-8, radiolysis was promoted in an acidified solution and prevented by addition of cysteine, glutathione and ascorbic acid. No correlation was found between radiolysis of DMNA and formation of nitrite and nitrate ions in acid or alkaline media. DMNA irradiated in homogenates of fish was considerably more stable than in aqueous solution. [From En summ.] JA

41

[Formation of dimethynitrosamine (DMNA) in sea foods. I. Effect of haemoglobin and ascorbate on DMNA formation in Alaska pollack roe.]
Tozawa, H.; Sato, M.
Bulletin of the Japanese Society of Scientific Fisheries [Nihon Suisan Gakkai-shi] **40** (4) 425-430 (1974) [16 ref. Ja, en] [Tokai Regional Fisheries Res. Lab., Kachidoki, Chuo-ku, Tokyo, Japan]

The effect of haemoglobin (Hb) and ascorbate on the formation of carcinogenic dimethylnitrosamine (DMNA) by the reaction between dimethylamine (DMA) and nitrite in salted Alaska Pollack roe was investigated. When blood or Hb of carp were added to Alaska pollack roe or to minced halibut flesh together with DMA and nitrate, the formation of DMNA was accelerated. The addition of ascorbate to Alaska pollack roe or halibut flesh containing DMA, nitrite and Hb decreased the amount of DMNA formed. However, when ascorbate was added to a halibut flesh sample without Hb there was no change in the amount of DMNA produced. It is concluded that the ascorbate inhibits the accelerative activity exhibited by the Hb, rather than the reaction between DMA and nitrite. [From En summ.] AA

42

Polarographic determination of the nitrosation products of creatine and creatinine.
Velisek, J.; Davidek, J.; Klein, S.
Zeitschrift für Lebensmittel-Untersuchung und -Forschung **155** (4) 203-208 (1974) [15 ref. En, de] [Dept. of Food Chem. and Analysis, Inst. of Chem. Tech., Prague, Czechoslovakia]

A polarographic method for simultaneous determination of nitrosation products of creatine (N-nitrososarcosine) and creatinine (creatine-5-oxime, 1-methyldantoin-5-oxime) in meat is described. 10g of meat is homogenized by grinding with 10% perchloric acid. The homogenate is then made up to 100 ml with perchloric acid and filtered; 20 ml of the filtrate is then neutralized with KOH, held for 30 min at 0°C and filtered. 2 ml of the filtrate + 1 ml methanol are then transferred to a volumetric flask and the vol. made up to 10 ml with 0.1N HCl. The dissolved O_2 is then separated, and the polarographic waves are recorded; the positive wave corresponds to reduction of the two oximes, the negative wave corresponds to reduction of N-nitrososarcosine. Similar polarographic measurements in Mc'Ilvain buffer (pH 12.0) give values for creatine-5-oxime. The concn. of 1-methylhydantoin-5-oxime can then be calculated by difference. $NaNO_2$ can interfere with measurements in 0.1 N HCl; this may be eliminated by addition of methanol, the resulting methyl nitrite being removed by bubbling N_2 through the sample. Detection limit is approx. 15 mg/100 g meat. Analytical error was 1.78% for N-nitrososarcosine, 1.63% for creatine-5-oxime and 1.37% for 1-methylhydantoin-5-oxime. AJDW

43

Rapid formation of carcinogenic N-nitrosamines by interaction of nitrite with fungicides derived from dithiocarbamic acid in vitro under simulated gastric conditions and in vivo in the rat stomach.
Eisenbrand, G.; Ungerer, O.; Preussmann, R
Food and Cosmetics Toxicology **12** (2) 229-232 (1974) [12 ref. 1 n] [Inst. für Toxikologie und Chemotherapie am Deutschen

Krebsforschungszentrum, 69 Heidelberg 1, Federal Republic of Germany]

Many agricultural chemicals contain structures that can be N-nitrosated and food may contain residues of such compounds in the presence of nitrite. The formation of the carcinogen dimethylnitrosamine (DMN) from the fungicide ziram (bis-(dimethylthiocarbamato)zinc) in the presence of sodium nitrite was studied. Under the selected conditions, optimum pH for the formation of DMN was 1.5-2.0; more than 1 mg DMN being produced after a 10 min incubation of 10^{-4} moles ziram with 20-fold molar excess of nitrite. Ferbam and DPTD (dipyrrolidylthiuram disulphide) produced DMN and N-nitrosopyrrolidine respectively under the same conditions. It is concluded that fungicides derived from dithiocarbamic acid rapidly form significant amounts of carcinogenic nitrosamines when they come into contact with nitrite under the conditions prevailing in the stomach. Residues of these fungicides, widely used in the cultivation of fruit and leafy vegetables, represent a potential starting material for the formation of carcinogenic nitrosamines when ingested with nitrite. VJG

44

Nitrosamines in search of tumours.
Anon.
Food and Cosmetics Toxicology **12** (2) 251-255 (1974) [En]

Induction of tumours by nitrosamines is considered under the following headings: carcinogenic activity of individual nitrosamines; targets in the nervous system for tumour production; and alkylation, immunosuppression and cancer. VJG

45

Formation of N-nitrosopyrrolidine from pyrrolidine ring containing compounds at elevated temperatures.
Huxel, E. T.; Scanlan, R. A.; Libbey, L. M.
Journal of Agricultural and Food Chemistry **22** (4) 698-700 (1974) [9 ref. En] [Dept. of Food Sci. and Tech., Oregon St. Univ., Corvallis, 97331, USA]

The formation of N-nitrosopyrrolidine from various pyrrolidine ring containing compounds and sodium nitrite at elevated temp. was investigated. N-Nitrosopyrrolidine was formed when dry samples of L-proline, glycyl-L-proline, L-prolylglycine, and pyrrolidine were heated with nitrite at 170°C for 2 h and when proline was heated for 2 h with sodium nitrite at 170°C in a pH 6.2 buffer solution. Buffered collagen samples produced N-nitrosopyrrolidine at temp. of 120°C and above. L-Hydroxyproline did not produce N-nitrosopyrrolidine. The identity of N-nitrosopyrrolidine was confirmed in the heated samples by GLC coupled with MS. AS

46

Further survey of cured meat products for volatile N-nitrosamines.
Panalaks, T.; Iyengar, J. R.; Donaldson, B. A.; Miles, W. F.; Sen, N. P.
Journal of the Association of Official Analytical Chemists **57** (4) 806-812 (1974) [16 ref. En] [Food Res. Lab., Health Protection Branch, Health and Welfare Canada, Ottawa, Ontario, Canada]

80 samples of various kinds of cured meat products were analyzed for volatile nitrosamines. Nitrosopyrrolidine (NPy) was determined by using TLC and simple dialkylnitrosamines were measured by using a GLC method with a Coulson electrolytic conductivity detector (pyrolytic mode). 17 samples contained 13-35 parts/billion NPy; 29 samples contained 2-35 parts/billion dimethylnitrosamine (DMN); 9 samples contained 2-25 parts/billion diethylnitrosmine (DEN). In a few cases the identities of NPy, DMN and DEN were confirmed by GLC-MS. AS

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